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(54)

**Fine polishing compound for wafer.**

(57)

A fine polishing compound for polishing a silicon wafer used as a substrate crystal in electrical integrated circuits, which comprises water, colloidal silica particles, a water-soluble polymeric compound, and a water-soluble salt.

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## Fine Polishing Compound for Wafer

Background of the Invention

This invention relates to a compound for smoothly polishing the surface of a wafer widely used as a substrate crystal for electrical integrated circuits, more specifically, to a fine polishing compound for smoothly polishing the surface of a wafer to remove irregularities greater than 10 mμ.

Wafers which are widely used as substrates for electrical integrated circuits are typically produced by slicing a silicon or germanium crystal ingot, and the surface of these wafers is polished to form a flat surface having as less irregularities as possible, which are then used for the formation of circuits.

This is because, if there are irregularities on the surface of the wafer when circuits patterns are depicted on the surface of the wafer, precise and fine depicting will become difficult, and uneven electrical characteristics of the wafer will result.

Heretofore, to polish the surface of such a wafer, there have been proposed a variety of polishing agents.

For example, the specification of U.S. Patent 3,170,273 discloses a silica sol containing 2 to 50% by weight of silica and a silica gel containing 2 to 100% of silica as polishing agents. The specification of U.S. Patent 3,328,141 discloses that when these polishing agents are mixed with an alkaline compound to adjust the pH value to 10.5 to 12.5, the resulting polishing agent have increased polishing speeds. However, observation by a differential interference microscope or the like of the surface of wafers polished using these polishing agents has revealed irregularities of 5 to 500 mμ, showing that these polishing agents are not very satisfactory.

Japanese Patent Publication No. 53-9910/1978 discloses a polishing agent containing quartz, silicates, and hexafluorosilicates, and further containing a monohydric alcohol having 3 to 5 carbon atoms, and polyvinyl alcohol. However, with such a polishing agent, a fully satisfactory surface has not yet been obtained, and a polishing agent containing these alcohols has a problem in that it is difficult to store the polishing agent for a long period of time.

Furthermore, Japanese Patent Publication No. 61-14655/1986 (corresponding to the specification of U.S. Patent 4,260,396) discloses a polishing agent containing water-soluble carboxymethylene rubber or xanthane gum. However, this polishing agent has problems in that it requires a long polishing time required to obtain a smooth polished surface and subsequently a long time for so-called after-polishing with water for cleaning. Moreover, it involves an increased minimal amount of silica required for the formation of a smooth surface, which is another disadvantage of this polishing agent.

With a view to eliminate the prior art problems of polishing compounds, it is a primary object of the present invention to provide:

1. a polishing agent which gives a polished surface having no irregularities detectable by a differential interference microscope,
2. a polishing agent which requires a short polishing time to obtain a smooth surface,
3. a polishing agent which requires a less amount of silica to obtain a smooth surface, and
4. a polishing agent which can be stably stored for a long period of time.

Summary of the Invention

The inventors have conducted intensive studies to solve the above problems. As a result, the inventors have obtained findings that the above object is attained by a compound which is based on a slurry containing colloidal silica particles, with a specific water-soluble polymeric compound and a specific water-soluble salt, and have accomplished the present invention.

In accordance with the present invention which attains the above object, there is provided a fine polishing compound for a wafer comprising water, colloidal silica particles, a water-soluble polymeric compound, and a water-soluble salt.

The present invention will now be described in detail. The colloidal silica useful for the present invention is referred to a stable colloidal dispersion of amorphous silica in a liquid. In this liquid, average particle diameter of the silica particles preferably ranges from 5 to 500 mμ, more preferably 100 mμ or less. If the average particles size is less than 5 mμ, ratios of silicic acid monomer and oligomer contained in the colloidal dispersion will become increased and, when the surface of a wafer is polished using such a polishing compound, silica tends to adhere to the surface of the wafer, which is undesirable. If the average

particle diameter exceeds 500 mμ, the surface of the wafer tends to be scratched, which is also undesirable.

The average particle diameter used in the present invention, when the particles are present separately with no agglomerations, means average particle diameter of the particles under that condition or, when the particles are agglomerated, means average particle diameter of the particles under that condition.

The colloidal silica useful for the present invention preferably has silanol on the surface of the particles in a surface density of 1 to 10 groups/nm<sup>2</sup>. When silanol on the surface of the particles has a surface density within the above range, hydrophilicity of the silica particles is improved, and the polishing compound has improved dispersibility in water compared with a polishing agent containing a silica powder which has silanol in a surface density of less than 1 group/nm<sup>2</sup>.

Surface density of silanol on the surface of the silica particles can be determined, for example, by the following method:

(1) First, silica sol is ion-exchanged to remove metal ions. Sodium hydroxide is added dropwise to the ion-exchanged silica sol (W g) to determine molar number (M) of sodium hydroxide required for the pH value to reach 8. Reaction in this case is considered to progress as follows: Si-OH + NaOH → Si-ONa + H<sub>2</sub>O

(2) Silica content (S weight %) of the ion-exchanged silica sol is determined by a drying method.

(3) Surface density of silanol is calculated by the following equation:

$$\begin{aligned}
 \text{Surface density (group/nm}^2\text{)} &= \frac{\text{Number of silanol in silica sol}}{\text{Surface area of silica in silica sol}} \\
 \text{Number of silanol in silica sol} &= M \times 6 \times 10^{23} \quad (\text{unit: group}) \\
 \text{Surface area of silica in silica sol} &= \frac{W \times S}{100 \times 2.2 \times 10^{-21}} \times \frac{4\pi r^2}{4\pi \cdot \frac{r^3}{3}} \\
 &= \frac{3}{220} W \times S \times \frac{10^{21}}{r} \quad (\text{unit: nm}^2) \\
 \text{Surface density (group/nm}^2\text{)} &= \frac{M \times 6 \times 10^{23}}{\frac{3}{220} W \times \frac{1}{r} \times 10^{21}} \\
 &= \frac{440 \times M \times r}{W \times S} \times 100
 \end{aligned}$$

wherein, r denotes an average particle radius (nm) of silica particles.

Colloidal silica having silanol in a surface density of 1 to 10 groups/nm<sup>2</sup> is obtained by subjecting water glass, with reduced content of sodium ions thereof, and silicic acid to a condensation polymerization reaction to form silica particles. Details of a production method for such colloidal particles are described in Chapter 3 of "The chemistry of silica: Solubility, polymerization, colloid and surface properties, and biochemistry" Ralph K. Iler JOHN WILEY & SONS(1979).

The polishing compound according to the present invention contains a water-soluble polymeric

compound. The polymeric compound, on polishing a wafer, serves to form a regular laminar flow between a polishing cloth and the surface of the wafer in the sliding direction of the polishing cloth to make the wafer surface even further smooth.

The water-soluble polymeric compound includes polymers of acrylamide, acrylic acid, methacrylic acid, itaconic acid, ethylene oxide, and vinylpyrrolidone, or copolymers of these monomers, copolymers of hydroxypropylcellulose and maleic acid with a vinyl monomer, guar gum, a graft copolymer of guar gum with a vinyl monomer, a graft copolymer of xanthane gum with a vinyl monomer, chondroitin sulfuric acid, hyaluronic acid, schizophyllan, mannan, and geran gum. Particularly preferable water-soluble polymeric compounds are polyacrylamide, a copolymer of acrylic acid and acrylamide, a graft copolymer of guar gum with acrylamide, and schizophyllan.

These compounds preferably have molecular weights of 100,000 or more. If the molecular weight is less than 100,000, a laminar flow is not formed between the polishing cloth and the surface of wafer during polishing, which is undesirable. Molecular weight of the polymeric compound used in the present invention is referred to that measured by a light scattering method (e.g., refer to Shin Jikken Kagaku Koza (New Experimental Chemistry), vol.19, p.571-585, published by Maruzen in 1978).

The water-soluble polymeric compound is prepared using the following procedures.

Polyacrylamide, a copolymer of acrylamide and acrylic acid, polyacrylic acid, polymethacrylic acid, polyitaconic acid, polymaleic acid, polyethylene oxide, and polyvinylpyrrolidone can be prepared by polymerization of the corresponding monomers using a conventional method known in the art.

Vinyl monomers which are copolymerizable with maleic acid include methylvinylether, ethylvinylether, and isobutylvinylether. Copolymers of maleic acid with these vinyl monomers can also be produced by a conventional method known in the art.

Guar gum is referred to viscous galactomannan contained in an albumen of seed of guar, a leguminous plant. Xanthane gum is referred to a polymeric polysaccharide obtained by pure cultivation fermentation of *Xanthomonas cansbestris*.

Vinyl monomers to be graft copolymerized with guar gum or xanthane gum include acrylic acid, methacrylic acid, styrenesulfonic acid, and acrylamide. One or more types of vinyl monomers can be graft copolymerized.

Graft copolymerization of the polymeric polysaccharide with vinyl monomers can be achieved using methods such as those described in detail in Journal of Applied Polymer Science, vol.30, 4013-4018 (1985) and vol.32, 6163-6176 (1986).

For example, when guar gum or xanthane gum is graft copolymerized with acrylamide, an aqueous solution containing 0.01 to 2.0 weight % of guar gum or xanthane gum is mixed with 1 millimole to 1 mole of acrylamide and 1 micromole to 10 millimole of a polymerization initiator per 1 g of guar gum of xanthane gum, and the mixture is reacted at a temperature of 0 to 100°C in the air or in an inert gas atmosphere. The polymerization initiator is preferably cerium ionic radicals, and it is preferable to use guar gum or xanthane gum as of higher purity as possible.

Chondroitin sulfuric acid is referred to chondroitin sulfuric acid which is a kind of mucopolysaccharide extracted from mammalian tela connectiva, or dermatan sulfuric acid. Hyaluronic acid is a kind of mucosaccharide extracted from a cockscomb. Schizophyllan is a polymeric polysaccharide extracted from *suehirotake*. Mannan is a polymeric polysaccharide obtained from endosperm cell wall of a palm or from marine algae such as *codium*. Geran gum is a polymeric polysaccharide produced by *Pseudomonas aerodae*.

Content of the water-soluble polymeric compound in the inventive polishing compound is typically 1 ppm or more and, specifically, preferably 10 to 1,000 ppm. When a wafer is polishing using a polishing compound having a content of the water-soluble polymeric compound within this range, a laminar flow is formed in the sliding direction between the polishing cloth and the wafer surface, thereby making the wafer surface even smoother. However, if the content is less than 1 ppm, it is difficult to form the laminar, and, if the content exceeds 1,000 ppm, a turbulent flow tends to be formed; both cases are undesirable.

The inventive polishing compound contains a water-soluble salt. The water-soluble salt has a function to reduce the thickness of hydration layer on the surface of silica particles in the slurry-formed fine polishing compound. This increases the kinetic action of the silica particles exerting on the wafer during polishing with the polishing compound, thereby increasing the mechanical polishing effect and improving the polishing efficiency.

The water-soluble salt includes, but is not restricted to, neutral salts comprising combinations of a cation selected from the group consisting of lithium, sodium, and potassium, and an anion selected from the group consisting of fluoride, chloride, bromide, iodide, sulfate, nitrate, and perchlorate. These water-soluble salts can be used alone or in combination of two or more types. Of these, potassium chloride and

potassium perchlorate are particularly preferable which are high in degree of ion dissociation and large in atomic radius of cation.

Content of the water-soluble salt in the inventive fine polishing compound for a wafer is typically 20 ppm or more. The thickness of hydration layer formed on the surface of silica particles can be reduced by the addition of the water-soluble salt, however, if the content is less than 20 ppm, a sufficient effect of the salt is not obtained, which is undesirable.

As needed, the inventive fine polishing compound for an wafer can be mixed with an alkaline compound to adjust the pH value of the compound to 8 to 12.

As the alkaline compound, hydroxide of an alkali metal, an amine, or ammonia can be used. The alkali metal hydroxide includes potassium hydroxide, sodium hydroxide, rubidium hydroxide, and cesium hydroxide. Of these, potassium hydroxide and sodium hydroxide are particularly preferable. The amine includes triethylamine, triethanolamine, monoethanolamine, diisopropanolamine, ethylenediamine, tetraethylpentamine, triethylpentamine, diethylenetriamine, and hexamethylenediamine. Of these, ethylenediamine is preferable.

The present invention has the following advantageous effects and is thus very important for use in industrial applications.

(1) When a wafer is polished using the inventive fine polishing compound, the water-soluble polymeric compound forms a laminar flow between the polishing cloth and the wafer, thereby preventing occurrence of a turbulent flow. Therefore, a smooth polished surface is obtained which has no irregularities detectable by a differential interference microscope.

(2) Since the inventive fine polishing compound contains colloidal silica as silica particles having on the surface silanol groups in a surface density of 1 to 10 groups/nm<sup>2</sup>, it has a high hydrophilicity of silica particles and an improved dispersibility in water, compared with a polishing agent which contains a silica powder containing silica particles having on the surface silanol groups in a density of 1 group/nm<sup>2</sup> or less.

(3) When a wafer is polished using the inventive fine polishing compound, because the thickness of hydration layer on the surface of colloidal silica particles in the slurry is small due to the function of the water-soluble salt contained in the compound, kinetic action of the silica particles exerted on the wafer when the silica particles impinge upon the wafer during polishing is increased, thereby increasing the mechanical polishing effect, and thus the fine polishing efficiency is improved over that for polishing with a compound which does not contain the salt.

#### Description of the Preferred Embodiments

The present invention will now be described further in detail with reference to examples and comparative examples, but these examples are not intended to limit the invention. Examples 1-15, Comparative Examples 1-2

#### (1) Preparation of guar gum graft-copolymerized with acrylamide

Polymeric guar gum (from Sansho, named Meipro Gut 120) was dissolved in water to obtain a 0.5% aqueous solution. The solution was purified using filtration and alcohol dissolution techniques. The purification was carried out using the following procedures:

First, using a mesh filter having a pore size of 1-10 microns, coarse impurities were removed from the guar gum aqueous solution by the filtration method. Since guar gum is insoluble in alcohol, the guar gum aqueous solution was poured into alcohol to dissolve alcohol-soluble substances, and the precipitated substance was retrieved. The precipitated substance was dried, and again dissolved in water to obtain a guar gum aqueous solution of a predetermined concentration, 1 weight % in this example.

1 liter of the thus obtained 1 weight % aqueous solution was mixed with 0.5 mole of acrylamide under stirring and purging with nitrogen, and then mixed with 0.3 millimole of cerium ammonium nitrate as a polymerization initiator and reacted for 24 hours at room temperature under further stirring to obtain acrylamide-grafted guar gum (hereinafter referred to as "GGG").

#### (2) Preparation of polishing compound

Colloidal silica having an average particle diameter of 60 millimicrons, containing 50 weight % of silica

particles, and having silanol on the surface of the particles in a surface density of 8 groups/nm<sup>2</sup>; GGG as the water-soluble polymeric compound, an aqueous solution (0.5 weight %) of polyacrylamide (from Mitsubishi Kasei, named Diaclear MN3000) (hereinafter referred to as "PAAM"), schizophyllan (hereinafter referred to as "SPH"); and KCl as the water-soluble salt were weighed in ratios shown in Table 1, and mixed with pure water to obtain polishing compounds containing 0.1 weight % colloidal silica. However, for Comparative Example 2, silica powder (from Nippon Silica Kogyo, named Nipseal E220A, having silanol in a surface density of 1 group/nm<sup>2</sup> on the particle surface) was dispersed in pure water in an amount of 1 weight %

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### (3) Fine polishing tests of wafer

Using the SPAW36 Polisher of Speed Fam and a soft, suede-type polishing cloth, a silicon wafer was polished while supplying the polishing compound at a rate of 1 liter per minute. Polishing pressure was 100 g/cm<sup>2</sup>, relative speed between the wafer and the polishing cloth was 1 m/sec, and temperature of the polishing cloth during polishing was 40°.

Time was determined until the surface of the wafer became smooth by polishing using the polisher (whether or not the surface was smooth was observed by a differential interference microscope in the course of polishing). The results are shown in Table 1.

After polishing by the polisher, the wafer was polished with a rinse and water in place of the polishing compound to remove silica and polymeric polysaccharides from the surface of the wafer. Time required for rinse polishing was determined (end point was determined by observation of the wafer surface with the differential interference microscope).

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### (4) Silica particle dispersion tests of polishing compounds

A slurry-formed fine polishing compound was put in a capped test tube of 5 cm in height and 100 cc in volume, which was allowed to stand for one day. The test tube was subjected to repeated cycles of turning upside down and then reverting back to the original position, and condition of silica particles uniformly dispersing in the slurry was observed visually to determine the number of cycles required for uniform dispersion.

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Table 1

Item No.	Water-soluble polymeric compound		Water-soluble salt		Alkaline substance		Water polishing test		Silica dispersion test of polishing compound
	Type	Content (ppm)	Type	Content (%)	Type	pH value of compound	Time for smooth surface	Rinse polishing time	
Example 1	GGG	100 *1	KCl	0.005	-	9.8	10 min	30 sec	5 cycle
2	GGG	100 *1	KCl	0.005	Ethylenediamine	11.0	9	30	
3	GGG	100 *1	KCl	0.01	-	9.7	5	30	
4	GGG	100 *1	KCl	0.05	-	9.6	3	30	
5	GGG	100 *1	KCl	0.1	-	9.6	1	30	
6	PAAM	100	KCl	0.005	-	9.8	5	30	
7	PAAM	100	KCl	0.005	Ethylenediamine	11.0	3	30	
8	PAAM	100	KCl	0.01	-	9.7	0	30	
9	PAAM	100	KCl	0.05	-	9.6	5	30	
10	PAAM	100	KCl	0.1	-	9.6	3	30	
11	SPH	100	KCl	0.005	-	9.8	0	30	
12	SPH	100	KCl	0.005	Ethylenediamine	11.0	9	30	
13	SPH	100	KCl	0.01	-	9.7	5	30	
14	SPH	100	KCl	0.05	-	9.6	3	30	
15	SPH	100	KCl	0.1	-	9.6	1	30	
Comparative Example 1	-	-	-	-	-	9.8	over 20min	-	5
2	GGG	100 *1	-	-	Ammonia	10.0	5	1 min	100

Note: \*1 Content converted to guar gum

The following can be seen from Table 1:

1. When a wafer is polished using the inventive polishing compound, in particular, using that containing large amounts of the water-soluble salt, fine polishing time is reduced and the polishing compound is suited for use as a polishing agent. Furthermore, silica particles of the slurry have good dispersibility, which makes the polishing compound easy to handle.

2. On the other hand, a polishing compound such as Comparative Example 1 which does not contain a water-soluble polymeric compound cannot achieve fine polishing. When silica particles are given in the form of a silica powder as in Comparative Example 2, the polishing compound is suitable for fine polishing, but is inferior in dispersibility to the inventive polishing compound and requires continuous stirring during polishing operation.

### Claims

1. A fine polishing compound for polishing a wafer comprising water, colloidal silica particles, a water-soluble polymeric compound, and a water-soluble salt.

2. The fine polishing compound for polishing a wafer as claimed in Claim 1 wherein said colloidal silica particles have an average particle diameter of 5 millimicrons to 500 millimicrons and on the surface thereof have silanol groups in a surface density of 1 to 10 groups/nm<sup>2</sup>.

3. The fine polishing compound for polishing a wafer as claimed in Claim 1 wherein said water-soluble polymeric compound has a molecular weight of 100,000 or more and comprises one or more types selected from the group consisting of polyethylene oxide, polyacrylamide, polyvinylpyrrolidone, hydroxypropylcellulose, polyacrylic acid, polymethacrylic acid, polyitaconic acid, polymaleic acid, a copolymer of maleic acid and a vinyl monomer, guar gum, a graft copolymer of guar gum with a vinyl monomer, a graft copolymer of xanthane gum with a vinyl monomer, chondroitin sulfuric acid, hyaluronic acid, schizophyllan, mannan, and geran gum.

4. The fine polishing compound for polishing a wafer as claimed in Claim 1 wherein said water-soluble polymeric compound is selected from the group consisting of polyacrylamide, a copolymer of acrylamide and acrylic acid, a graft copolymer of guar gum with acrylamide, and schizophyllan.

5. The fine polishing compound for polishing a wafer as claimed in Claim 1 wherein said water-soluble salt comprises one or more combinations of a cation selected from the group consisting of lithium, sodium, potassium, cesium, and ammonium, and an anion selected from the group consisting of fluoride, chloride, bromide, iodide, sulfate, nitrate, perchlorate, carbonate, formate, acetate, acrylate, and oxalate.

6. The fine polishing compound for polishing a wafer as claimed in Claim 1 wherein said water-soluble salt is selected from the group consisting of potassium chloride and potassium perchlorate.

7. The fine polishing compound for polishing a wafer as claimed in Claim 1 containing said colloidal silica in an amount of 0.1 weight %, said water-soluble polymeric compound in an amount of 1 ppm or more, and said water-soluble salt in an amount of 20 ppm or more.

8. The fine polishing compound for polishing a wafer as claimed in Claim 1 wherein pH value of said fine polishing compound is adjusted to 8 to 12.



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## EUROPEAN SEARCH REPORT

Application Number

EP 89 12 2550

### DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	PATENT ABSTRACTS OF JAPAN, vol. 10, no. 85 (C-336)[2142], 4th April 1986; & JP-A <sup>3</sup> 60 219 274 (DAINIPPON INK KAGAKU KOGYO K.K.) 01-11-1985 * Abstract *	1	C 09 G 1/02 H 01 L 21/306
X,P	EP-A-0 322 721 (MITSUBISHI MONSATO CHEM. CO.) * Claims 1-7 *	1,3-8	
A	FR-A-2 414 387 (W.R. GRACE) * Claim 1 * & US-A-4 260 396 (Cat. D)	1	
A	US-A-4 581 042 (G.A. WILLMORE) * Claims 1,7 *	1	
The present search report has been drawn up for all claims			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			C 09 G H 01 L
Place of search		Date of completion of search	Examiner
The Hague		18 November 91	BEYSS E.
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